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A RAPID RADIOCHEMICAL PROCEDURE FOR TIN

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#### ADMINISTRATIVE INFORMATION

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# ABSTRACT

A very rapid radiochemical procedure has been developed for the isolation of radioisotopes of tin from their fission-product isobars. An irradiated uranium solution containing tin and antimony carriers is added to a solution of sodium borohydride. The volatile stannane ( $\text{SnH}_4$ ) formed is decomposed in a hot quartz tube to the metal, which is collected on a cold surface. Stibine ( $\text{SbH}_3$ ), which is also formed under these conditions, is removed by absorption on an 'ASCARITE' column.

The tin chemical yield ranges between 15 % for an Sb decontamination factor of  $2 \times 10^4$  to 60 % for an Sb decontamination factor of  $10^3$ . The time required for separation of the tin metal from the other fission product elements is about 10 sec. Decontamination factors of other Sn descendents are: I =  $7 \times 10^4$ , and Te  $> 2 \times 10^4$ . Arsenic is also volatilized as the hydride; however, it is not necessary to eliminate it in this work for the determination of the Sn fission yield.

## SUMMARY

### Problem

A rapid radiochemical procedure for tin fission products does not exist. The problem was to develop a radiochemical procedure that would take less than one minute to separate tin from its precursor and descendant radionuclides, so that independent fission yields and radiation characteristics of tin isotopes could be determined.

### Findings

A 10-second radiochemical method was developed for the separation of tin fission-product isotopes from their parent and daughter radionuclides with decontamination factors  $> 10^4$ . It involves a distillation of tin hydride followed by thermal decomposition to the metallic state. Independent fission yields for  $\text{Sn}^{131}$ ,  $\text{Sn}^{132}$ , and  $\text{Sn}^{133}$  have been determined for thermal-neutron-irradiated  $\text{U}^{235}$ .

## INTRODUCTION

A very rapid radiochemical procedure for the separation of tin from mixed fission products (MFP) was required in order to determine the fission yields and radiation characteristics of short-lived tin isotopes. A simple one-step procedure was sought which would have the required speed and would give an exact time for the tin separation. The decontamination factors for antimony, tellurium and iodine had to be large since the fission yield of the tin nuclide is finally determined from the amount of a longer-lived radioactive iodine descendent arising from the decay of this separated tin.

The Nuclear Science Series Monograph, The Radiochemistry of Tin,<sup>1</sup> lists a number of radiochemical methods for separating tin from MFP. These procedures include separations by multiple sulfide precipitations, distillation of the halides, solvent extraction, and combinations of these methods. The time for separation varies from 20 min to 4 hr, and chemical yields vary from 50 to 95 %. Decontamination factors for descendent and parent elements are not given. Since these methods are unsatisfactory for studies of short-lived tin isotopes, other methods were investigated.

Production of tin hydride with an alkali borohydride has been reported.<sup>2-5</sup> This paper describes an application of these methods to give a rapid, clean radiochemical separation of tin from MFP. Slight modifications were made to this general tin procedure in order that irradiated samples could be handled rapidly for determination of tin fission yields.

## TIN SEPARATION PROCEDURE

### Reagents

The concentration of carriers and reagents used are: antimony (III), 10 mg Sb/ml in concentrated HCl; tin (IV), 400 mg Sn/ml in 3N HCl; and sodium borohydride solution, 120 mg  $\text{NaBH}_4$ /ml in 0.2N NaOH.

### Apparatus

The apparatus for the determination of tin is shown in Fig. 1. A simpler apparatus may be used if a rapid separation is not necessary. Unit A holds the solution of carriers, acid, and mixed fission products (MFP). It is fitted with a three-way stopcock to provide vacuum for introducing the solution of MFP into the vessel and nitrogen for forcing the solution rapidly into unit B. Unit A also has a side arm with a needle valve in series to introduce nitrogen for flushing the air out of the system before starting the reaction. This unit and the method of introducing the sample to it are the same as unit A described previously in the procedure for the rapid separation of antimony and arsenic hydrides from MFP.<sup>6</sup>

Stannane is formed in unit B, which is a pyrex tube of 18-ml volume with a ground female joint that fits onto section A. The gases from unit B pass to a calcium chloride drying tube, of 12 mm I.D. and 100 mm long, which contains "drierite" (8-mesh anhydrous  $\text{CaSO}_4$ ). This tube is fitted with a three-way stopcock, one leg of which is vented to a hood. The other leg is in series with a 100-ml syringe, unit C, fitted with a weighted piston to give a pressure of about one p.s.i.g. The syringe acts as a surge chamber to hold the rapidly evolved gas from unit B. A needle valve in line directly after unit C is used for controlling the flow rate of the gas through the rest of the system. Next, the gases pass through a pyrex tube, of 11 mm I.D. and 150 mm long, of which 110 mm is filled with "ascarite" (20-30 mesh sodium hydroxide-impregnated asbestos absorbent); a quartz tube heated by a Meker-type burner; and a bubbler to indicate the flow rate of the flushing gas. Rubber tubing carries the waste gas from the bubbler to the hood. Since

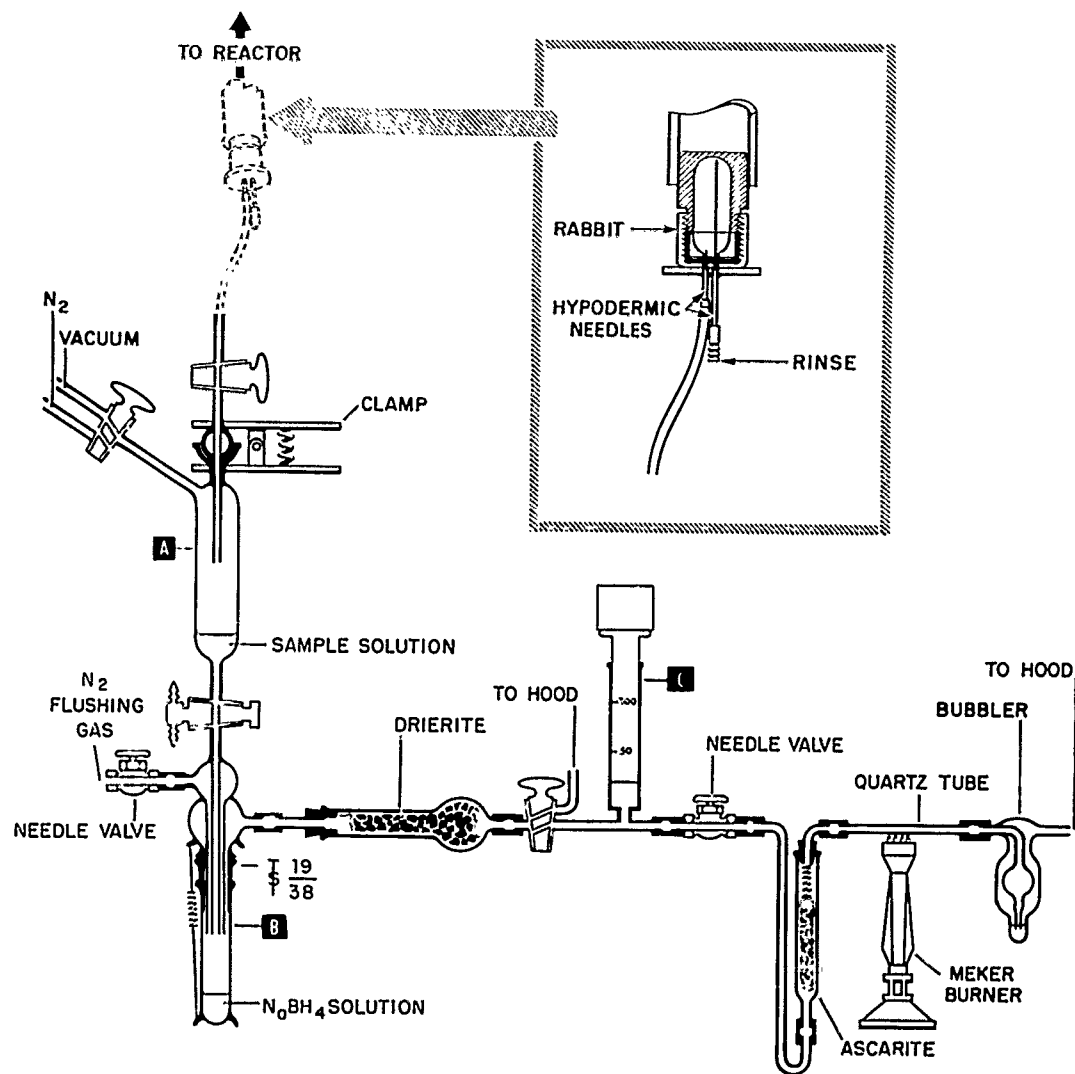


Fig. 1 Apparatus for Tin Separation

stibine and stannane are extremely toxic, it is imperative that they be vented to a hood and thus diluted with air to a non-toxic concentration.

#### Procedure

In Fig. 1 unit B contains one ml of sodium borohydride ( $\text{NaBH}_4$ ) solution. This unit and the train of apparatus following it is flushed for a few minutes with nitrogen before starting the analysis. The needle valve between unit C and the ascarite column is adjusted to a flow rate of 10 ml/sec. This adjustment is made in the following manner. With nitrogen flowing through the system, the rubber tubing leading out of the bubbler is pinched so that no gas escapes past the bubbler. The valve which lets the flushing gas into A is turned off when the piston of the syringe reaches the 100-ml graduation on the barrel. If the plunger remains stationary, the system is gas-tight. A stopwatch is started at the same time the gas is allowed to flow out of the bubbler. It should take the syringe 10 sec to empty. In a few trials the correct adjustment of the needle valve can be made. This adjustment should have to be made only once after the apparatus is assembled.

Unit A contains 1 ml of 0.6N HCl and 1 ml of Sb (III) carrier. The quartz tube is heated until it just starts to glow.\* The vacuum is applied (unit A) and the MFP solution containing 10 mg Sn (IV) carrier is drawn into unit A followed by 1 ml of 0.6N HCl rinse solution. The vacuum line is turned off, and the inert gas (under 2 lb pressure) is applied by a half-turn of the three-way stopcock. The reagents are then added to the  $\text{NaBH}_4$  solution in unit B. The tin metal deposits on the quartz tube within about 2 seconds from the start of the reaction. After 10 sec the three-way stopcock between the drierite and unit C is given a half-turn. This vents any residual gas to the hood so that the quartz tube with the tin deposit can be safely and quickly removed and that the time from the end of irradiation to the tin separation can be better known. The collected sample is dissolved from the tube in a closed system with cold 50 %  $\text{H}_2\text{SO}_4$  containing a carrier of the appropriate descendent element (iodine in the present studies). Other reagents may be used for this solution step or the radiations from the separated tin may be measured directly from the quartz tube depending upon the purpose of the analysis.

\*It is best to heat the central portion of the quartz tube just prior to use so the ends will remain cool. This prevents damage to the rubber tubing and provides a cool portion for the deposition of the tin mirror. While the temperature of the tube does not seem to be critical, overheating will cause apparent migration of the tin into the quartz.

$\text{Sn}^{113}$ , which is not formed in fission, may be used for the chemical yield determination if it is added with the tin carrier. An aliquot of the dissolved tin mirror is counted or it is assayed through gamma pulse-height analysis after the fission product tin and its descendents have decayed or have been otherwise separated. The  $\text{Sn}^{113}$  counting rate, after correcting for aliquot size and any decay of the  $\text{Sn}^{113}$ , is a measure of the chemical yield. Luke's<sup>7,8</sup> photometric method for the determination of tin in microgram amounts is an alternative method of finding the chemical yield.

Except for the small amount of gas vented to the hood, all fission product liquids and gases are retained within the apparatus. Since the hydrides of tin and antimony are extremely toxic, a fully enclosed system such as has been described, affords a maximum of safety.

## DEVELOPMENT OF TIN PROCEDURE

### Effect of Tin Oxidation State

Our experiments showed that the oxidation state of the tin carrier solution had no detectable effect on the production of stannane. This is not unexpected since  $\text{NaBH}_4$  is a very strong reducing agent. In the application of this procedure to independent fission yields, Sn (IV) was used because most of the fission-product tin is expected to be initially in its highest oxidation state, and stannous chloride is readily oxidized by the air<sup>9</sup> dissolved in irradiated solutions.

### Separation of Volatile Materials From Stibine and Stannane

A clean separation of tin from iodine was essential to determine the independent fission yields of Sn. The desiccant drierite, anhydrous calcium sulfate, which works well in removing iodine activities from stibine,<sup>6</sup> was used. It holds back other volatile fission product activities as well. A glass wool plug just before the desiccant stops most of the spray and enables one to make several runs before changing the desiccant. A small spring between the one-hole rubber stopper and the drierite maintains constant pressure on the column so that the gases cannot channel.

### Separation of Stibine From Stannane

While the reaction of a tin salt with sodium borohydride gives a good yield of stannane, stibine, which has similar physical properties, is also formed in significant amounts. The major effort in developing this procedure was directed toward a separation of the gases stannane and stibine. As mentioned previously, the quantity of iodine descendant is a measure of the cumulative tin yield. Therefore, a high decontamination from all elements in the mass chain was required. In previous work<sup>6</sup> it was found that arsine ( $\text{AsH}_3$ ) and stibine ( $\text{SbH}_3$ ) can be separated from each other by passing the mixture of gases through a quartz tube at  $600^\circ\text{C}$  where only the stibine is decomposed to the metal. The arsine passes through this tube and is decomposed to the metal in

a second quartz tube heated to a higher temperature. The contamination of antimony in the arsenic mirror was only 0.05 %. It was not possible to make a satisfactory separation of tin and antimony in this manner.

From Mellor<sup>10</sup> a list of possible materials which would preferentially absorb stibine from a stibine-stannane mixture was compiled and studied. At the high flow rates needed to give a rapid separation of the tin from MFP, liquid traps were ineffective. The gas was not scrubbed well enough to reduce the stibine contamination to an acceptable level. Solid traps found to retain varying amounts of stibine were: mercurous salts, freshly reduced copper powder, silver nitrate, potassium hydroxide, and sodium hydroxide. These solids were mixed about 1:1 with asbestos in order to maintain a fast flow of gas through the absorbant. Of these reagents, potassium and sodium hydroxide gave the best separation of the hydrides under the experimental conditions established. Since these hydroxides are hygroscopic and difficult to handle, a commercially available mixture of NaOH and asbestos, developed by J. B. Stetser<sup>11</sup> and called ascarite, was used. A trap 11 mm in diameter and 80 mm long passed only 0.039 % of the original amount of stibine entering. The amount of antimony holdback carrier was 1 mg. Figure 2 illustrates the absorption of stannane and stibine individually on an ascarite trap. Initially 1 mg of tin carrier was used in this separation since Schaeffer and Emilius<sup>3</sup> have shown that the tin yield has an inverse dependence upon the concentration of tin in solution. From Fig. 2 it can be seen that by increasing the tin concentration a better relative separation of stannane from stibine can be obtained. These curves show the absorption of the independent gases. With a mixture of stannane and stibine, the absorption behavior is somewhat different. When 33 mg of Sn (IV) carrier was used with 1 mg of Sb (III) carrier, more stibine came through the ascarite than when 11 mg of Sn (IV) was used with 1 mg of Sb (III).<sup>\*</sup> In the procedure adopted, 10 mg of Sn (IV) carrier is used with 1 mg of Sb (III) holdback carrier.

In order to get a still better decontamination from antimony, the column length was increased to 11 cm with a resultant sacrifice in tin yield. The ascarite column was filled in the following manner: (1) a plug of glass wool was placed in the constricted end of the 11 mm I. D. glass tube; (2) the ascarite was poured into the column up to a mark 11 cm from the glass wool plug with gentle tapping to settle the ascarite; (3) a short glass wool plug was placed over the ascarite filling; (4) a small steel compression spring was placed between the glass wool plug and the one-hole rubber stopper to prevent channeling.

<sup>\*</sup>One mg of Sb (III) carrier was used because with only a  $\mu$ g of Sb (III) carrier a quarter of a percent of the antimony activity deposited with the tin, and with larger amounts of Sb (III) carrier the ascarite column might be overloaded.

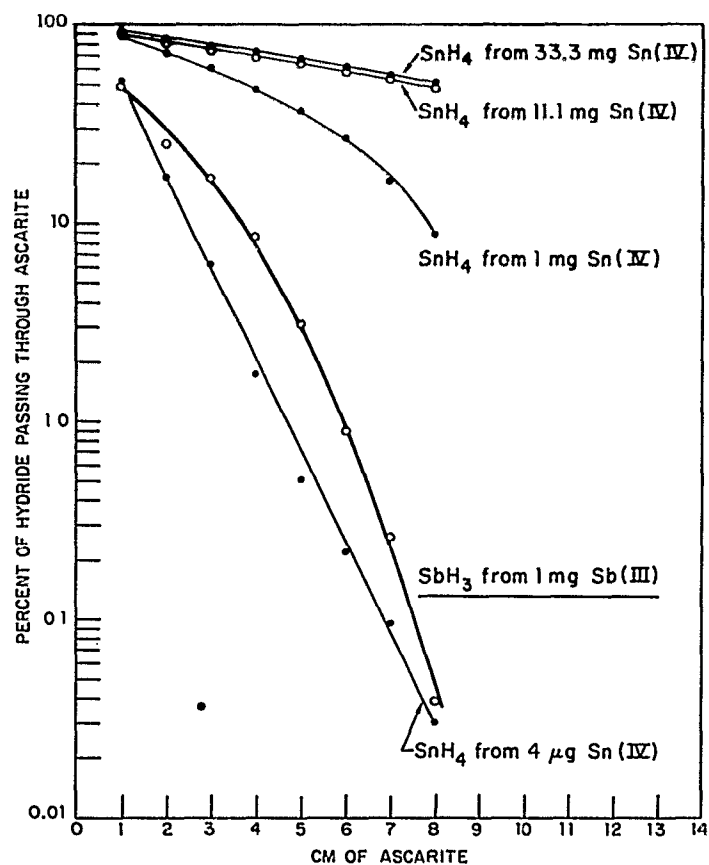


Fig. 2 Absorption of Stannane and Stibine on an Ascarite Trap

#### DETERMINATION OF TIN FISSION YIELDS

A specific application of the rapid separation of tin from its fission product descendants has been made in the determination of the fission yields of Sn-131, 132 and 133 in the thermal neutron irradiation of U<sup>235</sup>. Use was made of the same pneumatic system mentioned in a previous paper.<sup>12</sup> After leaving the reactor, the pneumatic sample carrier containing the MFP solution, 10 mg of Sn (IV) carrier, and Sn<sup>113</sup> tracer (for chemical yield determination) impales itself upon two hypodermic needles. The solution is drawn through a hypodermic needle by vacuum into A (Fig. 1), which contains 1 ml of 0.6N HCl and 1 mg of Sb (III) carrier. The rabbit and needle assembly are then quickly rinsed with 1 ml of 0.6N HCl, which is added through the second hypodermic needle. Emptying and rinsing the rabbit takes about 2 sec. The tin fission products are then separated as described in the tin procedure.

In the determination of the independent fission yield of a given tin isotope the separated tin was allowed to decay to its known iodine descendent. The tin was then dissolved in cold 50 % H<sub>2</sub>SO<sub>4</sub> containing a measured amount of iodine carrier, and a radiochemical determination of iodine was carried out. The amount of iodine found is a direct measure of the amount of tin originally present. The chemical yield of tin (which varied between 10 and 14 %) was determined by counting the added Sn<sup>113</sup> tracer after allowing the fission product tin and its daughters to decay to an insignificant level of activity.

It has been possible by the above technique to determine independent fission yields of tin fission products in thermal neutron fission of U<sup>235</sup>, tin half-lives, generic relationships, and also prominent gamma photopeak energies from pulse-height distributions taken of the rapidly separated tin fractions. The following results have been obtained from many different tin separations made at times after fission varying from 10 sec to 30 min:

<u>Nuclide</u>	<u>Half Life*</u>	<u>Fission Yield</u>
Sn <sup>131</sup> **	65 $\pm$ 10 secs	0.7 %
Sn <sup>132</sup>	50 $\pm$ 10 secs	0.5 %
Sn <sup>133</sup>	39 $\pm$ 15 secs	0.2 %

\* Independent experimental values of these Sn half lives were obtained through continuous evolution of SnH<sub>4</sub> from a basic NaBH<sub>4</sub> solution of U<sup>235</sup> during neutron irradiation and collection of the Sn descendents. on a charged wire. Tentative values by this method are 44 sec for Sn<sup>131</sup>, 50 sec for Sn<sup>132</sup>, and 43 sec for Sn<sup>133</sup>.

\*\*The 1.6 h isomer is not observed. Its fission yield would be less than 0.03 %.

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<p>Naval Radiological Defense Laboratory USNRDL-TR-632 A RAPID RADIOCHEMICAL PROCEDURE FOR TIN by A. E. Greendale and D. Love 28 February 1963 19 p. illus. 12 refs. UNCLASSIFIED</p> <p>A very rapid radiochemical procedure has been developed for the isolation of radioisotopes of tin from their fission-product isobars. An irradiated uranium solution containing tin and antimony carriers is added to a solution of sodium borohydride. The volatile (over)</p>	<p>Naval Radiological Defense Laboratory USNRDL-TR-632 A RAPID RADIOCHEMICAL PROCEDURE FOR TIN by A. E. Greendale and D. Love 28 February 1963 19 p. illus. 12 refs. UNCLASSIFIED</p> <p>A very rapid radiochemical procedure has been developed for the isolation of radioisotopes of tin from their fission-product isobars. An irradiated uranium solution containing tin and antimony carriers is added to a solution of sodium borohydride. The volatile (over)</p>	<p>1. Tin. 2. Isotope separation. 3. Radiation chemistry. I. Greendale, A. II. Love, D. III. Title. UNCLASSIFIED</p>
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